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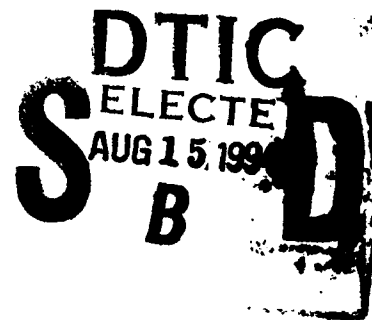


Thin Lithium Cobalt Dioxide Rechargeable Cells Using
Polyacrylonitrile-Based Polymer Electrolytes

Steve Slane

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13. ABSTRACT (Maximum 200 words) Rechargeable Li/LiCoO ₂ cells with polymer electrolytes have achieved 100 mAh/g capacity and over 75 charge/discharge cycles with an average discharge potential of 3.7 volts. Solid-state polymer lithium electrolytes based on poly(acrylonitrile) (PAN) have achieved room temperature conductivities of 1x10 ⁻³ siemens per cm, equal to that of some liquid organic electrolytes. Polymer films of ethylene carbonate, propylene carbonate, PAN, and lithium salts have yielded conductivities as high as 4x10 ⁻⁴ siemens per cm at 25 deg C. These high conductivities made the use of polymer electrolytes a viable possibility in advanced lithium batteries. Reported here are the film preparation techniques, conductivities from -70 to 70 deg C, and discharge curves of Li/LiCoO ₂ cells.				
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INTRODUCTION

The use of solid polymer electrolytes (SPE's) with a high energy cathode material such as LiCoO_2 in rechargeable lithium batteries is currently being widely investigated for both commercial and military applications. Polymer electrolytes can be prepared into very thin films possessing large surface area yielding high power densities. An energy advantage of a solid flexible electrolyte is its ability to enable the design of more volume efficient battery configurations. In an electrochemical cell, especially in a reversible cell, a flexible electrolyte can accommodate the volume changes that occur with charge/discharge cycles. A solid electrolyte can enable the development of bipolar batteries without intercell current leakage. Polymer electrolytes may also increase cell safety by preventing ignition by acting as a shutdown separator, if thermal runaway should occur within a cell. A thin Li^+ ion conducting polymer film acts as both the electrolyte and a separator between the lithium anode and a lithium insertion cathode. The use of high energy cathode films of reversible compounds such as LiCoO_2 , LiNiO_2 , LiMn_2O_4 , V_2O_5 , or V_6O_{13} with thin lithium foil anodes and the structural flexibility of polymer electrolytes makes the lithium polymer battery a promising candidate for advanced battery systems for electric vehicles (EV) or consumer/military electronics applications.

The use of Li_xCoO_2 ($0 < x < 1$) as a lithium intercalating electrode was first reported by Mizushima *et al.* (1). LiCoO_2 has a layered rock-salt structure and can theoretically deintercalate one mole of lithium per mole of oxide. In practice, the reversible range at ambient temperatures is ($1 < x < 0.5$) in Li_xCoO_2 . Due to the high Li^+ ion mobility in LiCoO_2 , $\text{Li}/\text{organic electrolyte}/\text{LiCoO}_2$ cells can be cycled more than 100 times at current densities of 1 to 10 mA/cm^2 with average discharge potentials of 3.7 to 3.9 volts.(2,3)

One of the first polymer electrolyte chemistries consisted of poly(ethylene oxide) PEO-LiX complexes (4,5) which need to operate at around 100°C . New multiphase systems involve adding plasticizing solvents to PEO-LiX or trapping liquid electrolyte solutions in a polymer matrix to form a "gel" electrolyte. The latter more "liquid-like" chemistries can operate at room temperature and therefore are of interest for further research. One of the basic fundamental problems in the development of solid state ionic materials based on polymers is the conductivities of these materials. Recently, room temperature conductivities as high as $10^{-3} \text{ S cm}^{-1}$ have been reported by Abraham and Alamgir (6) for poly(acrylonitrile) (PAN) based lithium salt complexes. It is this gel electrolyte chemistry utilized in Li/LiCoO_2 laboratory cells that has been investigated.

EXPERIMENTAL

The preparation of the solid gel electrolytes involved the immobilization of LiX in ethylene carbonate (EC) and propylene carbonate (PC) mixtures with PAN. The LiAsF_6 (Lithco "Lectro-salt") and $\text{LiN}(\text{CF}_3\text{SO}_2)_2$ (3M) were dried under vacuum at 60°C for 24 h. LiClO_4 (Alfa reagent grade) was recrystallized in distilled water then dried under vacuum at 150°C for 24 h. PC (Burdick and Jackson) was dried with type 4A molecular sieves for 48 h then distilled under vacuum. EC (Fluka AG) was fractionated under vacuum. Dimethyl carbonate (DMC) (Burdick and Jackson) was fractionated in an argon atmosphere. Karl Fisher titration for EC, PC, and DMC indicated water contents of < 24 ppm. Poly(acrylonitrile) (Polyscience Inc.) with an average molecular weight of 150,000 was dried under vacuum at 60°C for 48 h. Molecular sieves, type 3A, were ball-milled for 24 h then dried under vacuum at 60°C for 24 h.

The liquid electrolyte EC:PC:LiX was prepared in a vial with a stirring bar. PAN powder was then added and the mixture stirred to ensure wetting of the PAN. The mixture was heated slowly in an oil bath to 100°C , avoiding overheating and decomposing the PAN. The mixture turned to a clear highly viscous gel and was cast between glass plates, with 0.25 mm spacers, and allowed to cool. The resulting polymer electrolyte was an elastomeric mechanically stable film. Two general film compositions were prepared with mole percentages of 40EC:34.75PC:21PAN:4.25LiAsF₆ (17.6:1 EC + PC:LiAsF₆) and 38EC:33PC:21PAN:8LiAsF₆ (8.8:1 EC + PC:LiAsF₆). Variations on the 17.6:1 film include the addition of 5 weight percent molecular sieves, the mixture of DMC with EC and PC, and the reduction of PAN content to 12 mole percent. Chemical storage, film casting, and cell assemblies were performed in a Vacuum Atmospheres Company argon-filled dry box.

Electrolyte conductivities were determined from ac impedance measurements using an EG&G PAR model 388 impedance system with a frequency range of 5 Hz to 1000 kHz. The conductivity measurements were performed with a test cell with an electrode configuration of SS/SPE/SS. A thermocouple was in close proximity to the SPE in the cell. The cell assembly was inserted into a wide-mouthed glass reaction vessel packed with molecular sieves. Argon was bubbled through the vessel. The temperature testing (70°C to -70°C) was performed in a Tenney environmental chamber.

Thin film electrodes of LiCoO_2 were prepared by a modified spray sol-gel method which involved the thermal decomposition of organometallic starting materials on a hot aluminum substrate (7). Resulting film thickness was approximately 2 μm . X-ray diffraction confirmed the product to be LiCoO_2 . Due to the very thin nature of the electrode, no electrolyte was required in the cathode

bulk. Lithium, 5 mil, was used as the anode. The cell stack of LiCoO_2 / electrolyte / Li was sandwiched between stainless steel current collectors in a Teflon screw type cell. Electrode areas were approximately 0.7 cm^2 . They were cycled with ECO galvanostats between 4.2 and 3.25 volts at 25°C and the data collected with a Nicolet model 310 digital oscilloscope.

RESULTS AND DISCUSSION

For polymer electrolytes to be of practical use, Li-ion mobility must be high enough to enable useful rate capabilities in lithium batteries. The PAN-based electrolyte films demonstrate conductivities approaching that of the liquid and a significant increase in ion mobility over the PEO-based electrolytes. It is this result, first demonstrated by Abraham and Alamgir for this chemistry (6), that makes a mechanically stable free standing film a possible battery electrolyte. Having established that PAN-based films can be prepared with PC, EC mixtures and produce ionic conductivities close to liquid organic electrolytes, further studies of Li-ion mobility in these types of electrolytes were performed.

The arrhenius plots in Figure 1 show the conductivities of three LiX salt complexes, LiAsF_6 and LiClO_4 with the composition 40EC:34.75PC:21PAN:4.25LiX and $\text{LiN}(\text{CF}_3\text{SO}_2)_2$ with a composition of 56.5EC:23PC:16PAN:4.5LiN(CF_3SO_2)₂. The solvent to LiX ratio in all three electrolytes is 17.6:1. As in liquid organic systems the LiAsF_6 electrolyte produced the highest conductivity. The imide salt, known as a high temperature stabilizing salt, demonstrated poor conductivity at low temperatures. This is believed to be due to precipitation of the salt from the EC:PC mixture resulting in loss of lithium ions. In Figure 2, ionic conductivities of three compositional variations of the EC:PC: PAN:LiAsF₆ electrolyte are shown. Changing the solvent to LiAsF₆ ratio from 17.6:1 to 8.8:1 (i.e., doubling the amount of salt while holding the PAN concentration at 21 mole percent) lowers the conductivity over the whole temperature range. Lowering the amount of PAN from 21 mole percent to 12 mole percent increased the conductivity at lower temperatures. This is expected since the film with less polymer is more liquid-like. This "wetter" electrolyte still had the physical integrity of a solid film. Both these effects on ionic mobility are due to viscosity.

In Figure 3, a cyclic voltammagram of the second cycle, with a sweep rate of 2 mV/s, demonstrates the electrochemical window between 2.5 and 4.3 volts. This is a typical Li/LiCoO₂ voltammagram, with the cell achieving approximately 0.45 F/mol on charge and discharge. The dotted line on the graph represents a sweep of Li/EC:PC: PAN:LiAsF₆/Al resulting in negligible current in the voltage range of 2.0 to 4.5 volts. It can be concluded from these voltammagrams that this polymer electrolyte is stable in the operating voltages of a Li/LiCoO₂ cell.

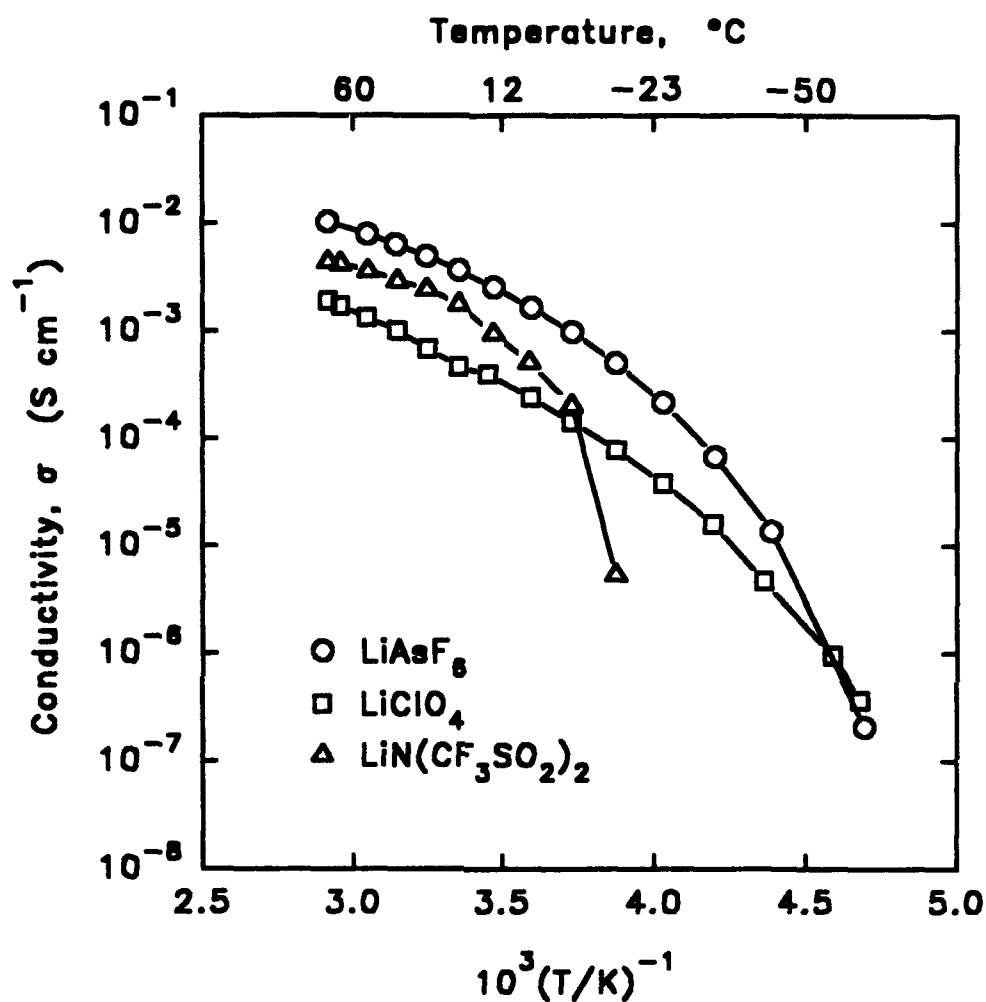


Figure 1. Arrhenius plot of conductivities of EC:PC:PAN:LiX electrolytes where EC+PC:LiX is 17.6:1.

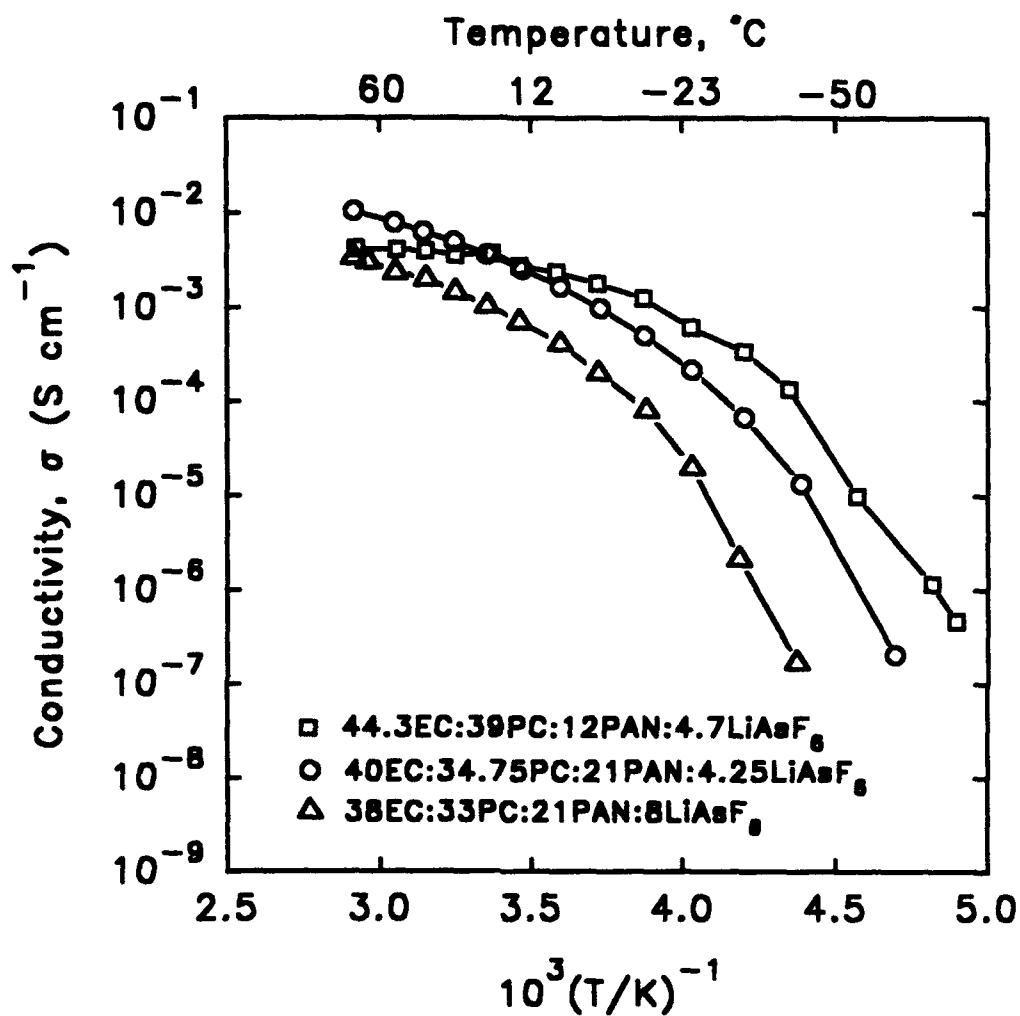


Figure 2. Arrhenius plot of EC:PC: PAN:LiAsF₆ electrolyte conductivities.

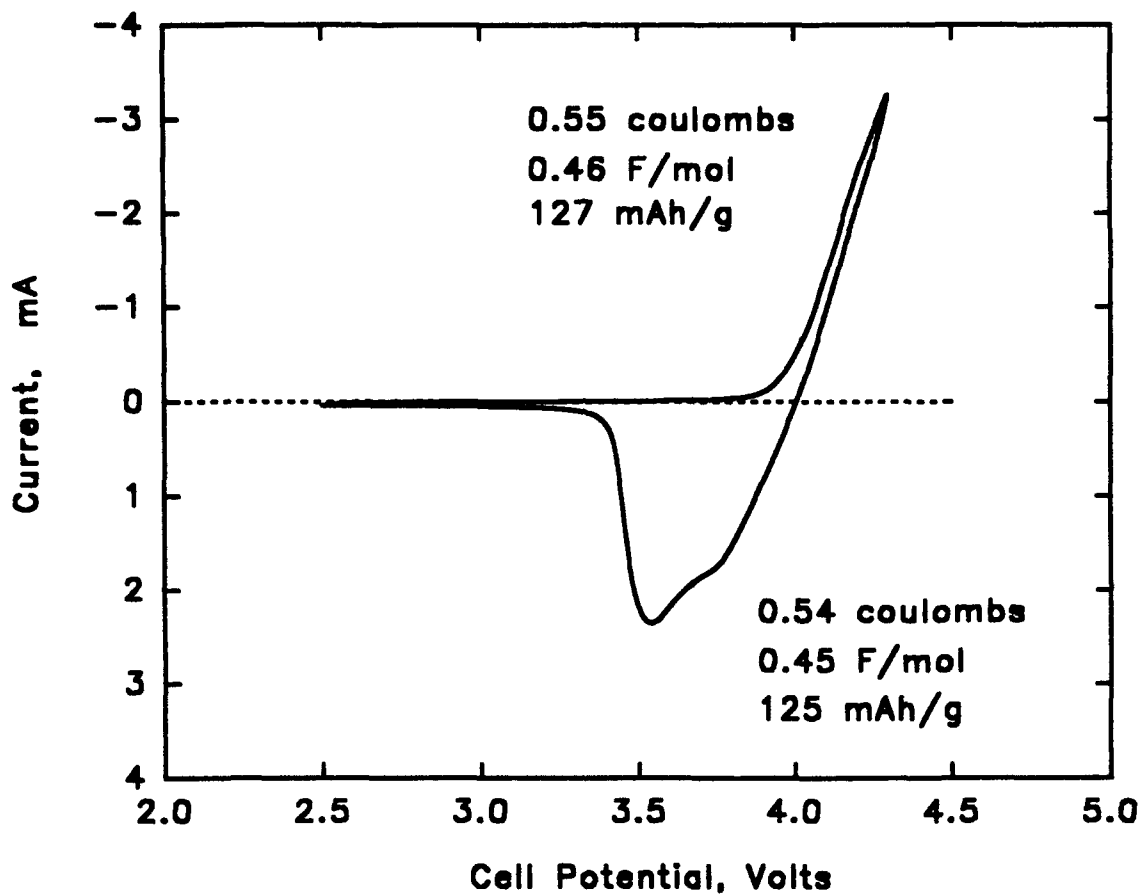


Figure 3. Cyclic voltammagram of Li/EC:PC: PAN:LiAsF₆/LiCoO₂ cell with 1.2 coulomb (277 mAh/g) capacity.

Discharge curves for a cell with 40EC:34.75PC:21PAN:LiAsF₆ electrolyte are displayed in Figure 4. The figure shows that the cell loses half its capacity in fifty cycles. Capacities of cells with other polymer electrolyte chemistries are shown in Table 1. Changing the solvent to salt ratio to 8.8:1 did not improve cycling. The addition of DMC, in liquid organic electrolytes makes the solvent more stable with lithium metal and improves lithium cycling efficiency (3). In the polymer the addition of DMC (35EC:30PC:9.75DMC:21PAN:4.25LiAsF₆) lowers the conductivity at lower temperatures due to its high melting point but the room temperature charge/discharge behavior is not as good as the film without DMC. The addition of ceramic materials such as γ -LiAlO₂ dispersed in PEO-based polymer electrolytes has been reported to increase ionic conductivity and reduce interfacial corrosion (8). It is believed the LiAlO₂ particles decreased the polymer crystallinity and absorbed impurities such as water that are reactive with lithium. In this study, type 3A molecular sieves were ground and dispersed in a mixture of 40EC:34.75PC:21PAN:4.25LiAsF₆ in the amount of 5 weight percent. The effect on conductivity was to lower slightly the ionic mobility at higher temperatures. Cells with this composite film also did not cycle as well as the baseline 17.6 :1 electrolyte. In general, the change in salt concentration, the addition of DMC, and the composite film formation with the sieves had little effect on conductivity at room temperature and above and no enhanced cycling performance was observed.

TABLE 1. Li/LiCoO₂ Discharge Capacities at 0.25 mA/cm² and 25°C.

ELECTROLYTE	CYCLE:	F/mol			
		1	25	50	100
EC:PC:PAN:LiAsF ₆ (21%PAN)		0.46	0.32	0.24	
EC:PC:DMC:PAN:LiAsF ₆		0.48	0.35	0.26	
EC:PC:PAN:LiAsF ₆ w/3A sieves		0.44	0.36	0.27	
EC:PC:PAN:LiAsF ₆ (12%PAN)		0.48	0.46	0.42	0.36

A significant improvement in cycling capacity is observed with the 12 mole percent PAN film (44.3EC:39PC:12PAN:4.7LiAsF₆). In Figure 5, a cell after 50 charge/discharge cycles is still delivering 0.42 F/mol and at 100 cycles only a 25% capacity loss is observed, both with excellent voltage retention. In Figure 6, the effect of current density on the discharge profile is shown. Even at 2 mA/cm² only a small loss in capacity and a slightly lower operating voltage is observed.

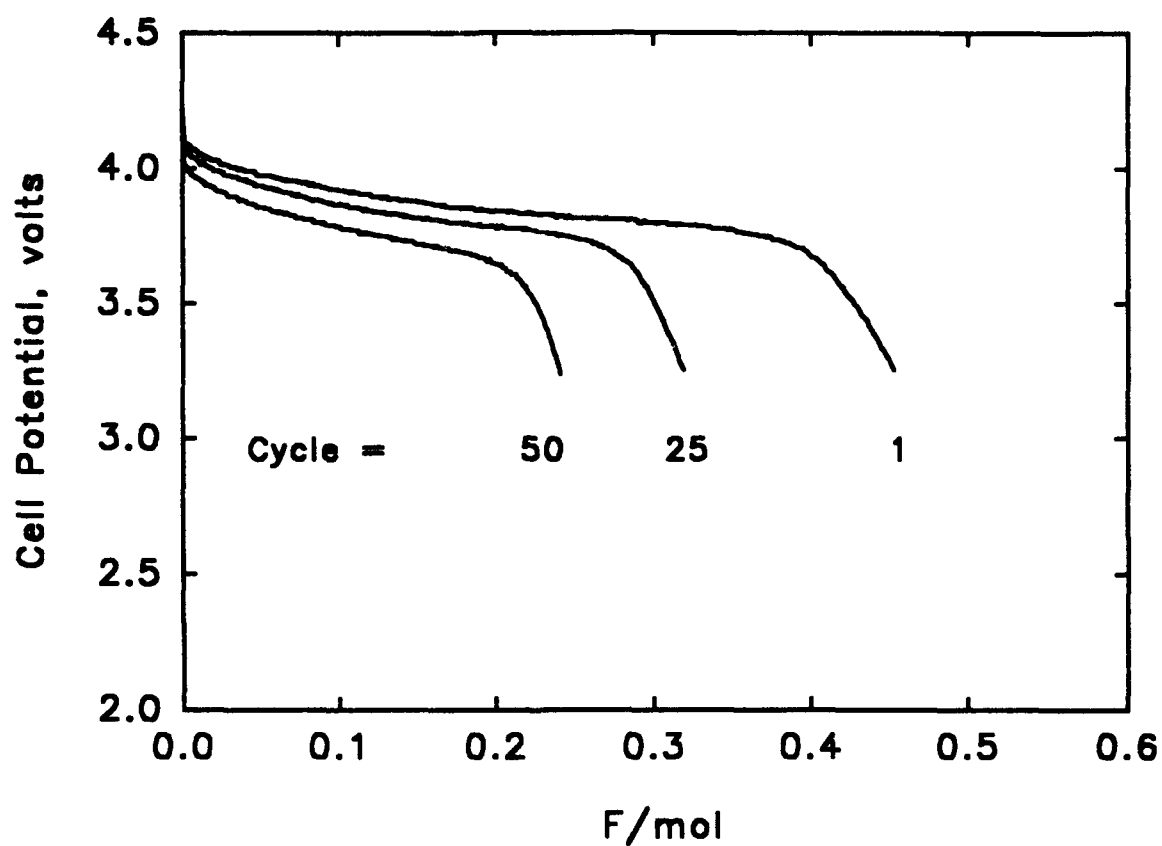


Figure 4. Discharge curves of a Li/LiCoO₂ cell with 40EC:34.75PC:21PAN:4.25LiAsF₆, charged and discharged at 0.25 mA/cm².

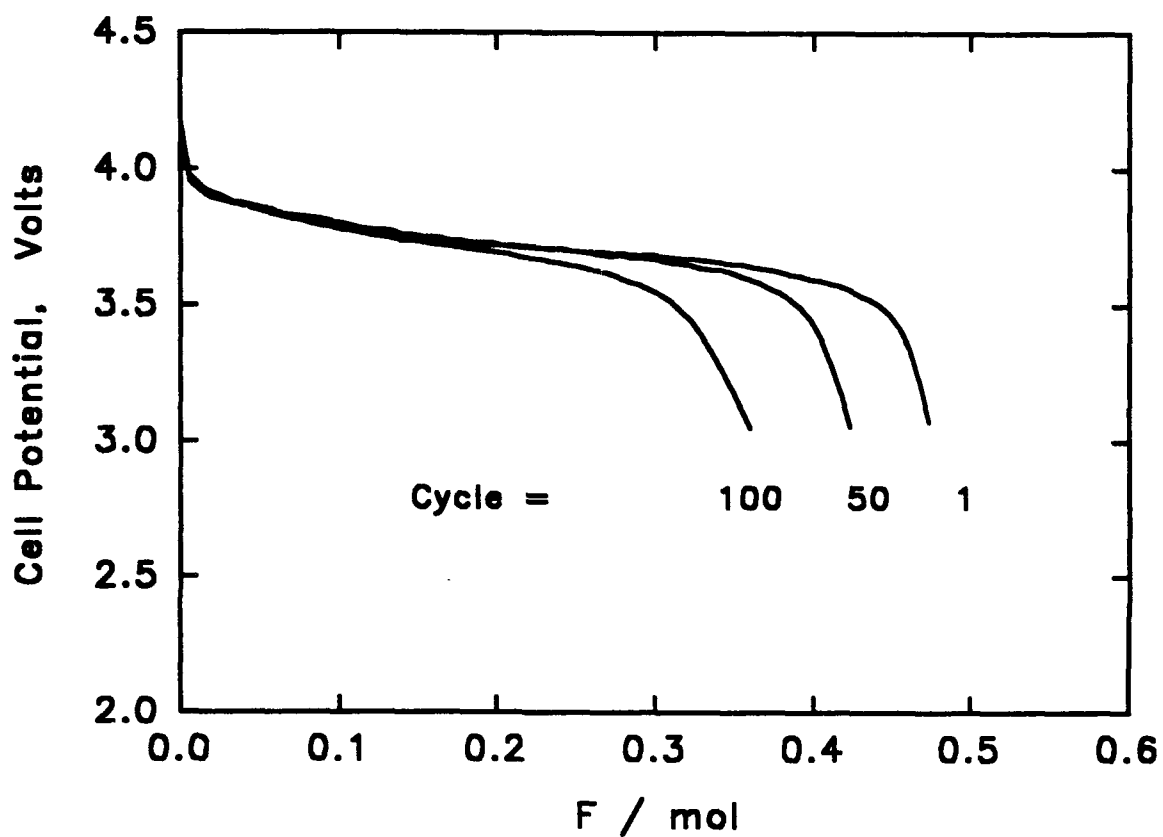


Figure 5. Discharge curves of a Li/LiCoO₂ cell with 44.3EC:39PC:12PAN:4.7LiAsF₆, charged and discharged at 0.25 mA/cm².

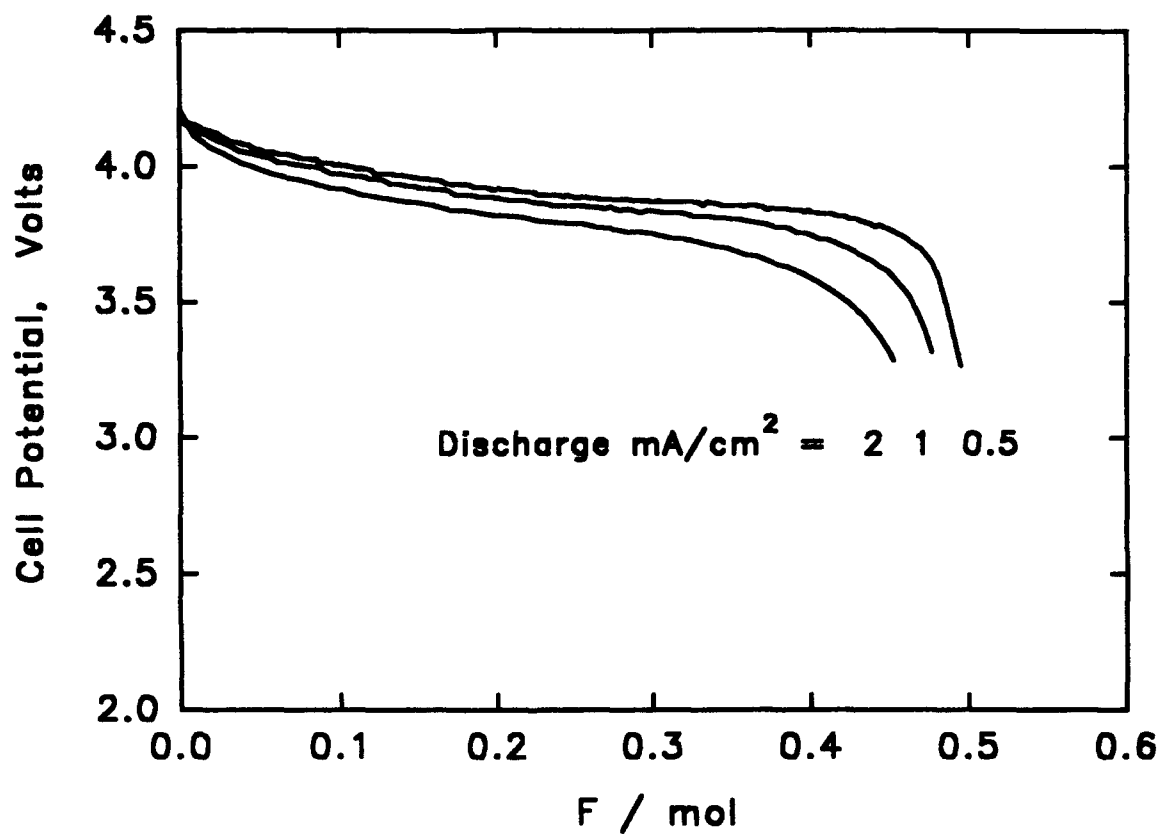


Figure 6. Discharge curves at various rates of a Li/LiCoO₂ cell with 44.3EC:39PC:12PAN:4.7LiAsF₆, charged. at 0.5 mA/cm².

CONCLUSIONS

It can be concluded from this study that solid gel polymers based on PAN and LiX salt complexes have adequate ionic conductivities to be used in lithium rechargeable batteries. Cycle performance was best with the electrolyte film that contained less PAN (12%). While improving conductivity and cycling, this film still maintained the physical integrity of a solid film. Future work will concentrate on improved electrolytes and thicker (greater capacity) cathodes which include polymer electrolyte in the composition.

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